Construction and Building Materials 172 (2018) 448–456

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Preparation, characterization, and determination of mechanical and thermal stability of natural zeolite-based foamed geopolymers

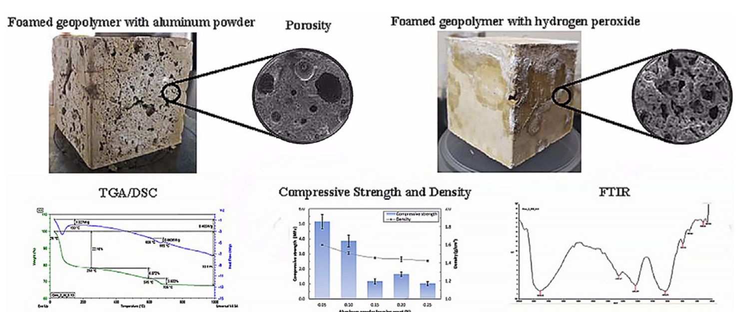
José Luis Villalba Lynch [a], Haci Baykara [a,b,c,*], Mauricio Cornejo [b], Guillermo Soriano [a], Néstor A. Ulloa [a,d]

[a] Facultad de Ingeniería Mecánica y Ciencias de la Producción, Escuela Superior Politécnica de Litoral, ESPOL, Campus Gustavo Galindo km 30.5 Vía Perimetral, Guayaquil, Ecuador
[b] Center of Nanotechnology Research and Development (CIDNA), Escuela Superior Politécnica de Litoral, ESPOL, Campus Gustavo Galindo km 30.5 Vía Perimetral, Guayaquil, Ecuador
[c] Departamento Ciencies Quimicas y Ambientales, Facultad de Ciencias Naturales y Matemáticas, Escuela Superior Politécnica de Litoral, ESPOL, Campus Gustavo Galindo km 30.5 Vía Perimetral, Guayaquil, Ecuador
[d] Facultad de Ingeniería Mecánica, Escuela Superior Politécnica de Chimborazo, ESPOCH, Panamericana Sur km 1½, Riobamba, Ecuador

HIGHLIGHTS

- Foamed geopolymers were prepared for the first time by the alkaline activation of Ecuadorian natural zeolite.
- Different amounts of Al powder and H_2O_2 (30% water solution) were used as foaming agents.
- The compressive strength results met Ecuadorian technical standards NTE INEN 643 and NTE INEN 638.
- Potential isolation materials were prepared for construction industry.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 19 September 2017
Received in revised form 27 March 2018
Accepted 27 March 2018

Keywords:
Foamed geopolymer
Ecuadorian natural zeolite
Potential Isolation materials
Aluminum powder
Hydrogen peroxide

ABSTRACT

This study is the first attempt to evaluate the effect of two foaming agents, aluminum powder and hydrogen peroxide (30 wt% water solution) on zeolite-based geopolymers and their possible use as building material according to Ecuadorian technical standards. These foamed geopolymers were prepared by alkali activation of an Ecuadorian natural zeolitic tuff and by alkali activation using NaOH (10 M), $Ca(OH)_2$ and $Na_4Si_5O_{12}$, with the use of two different foaming agents mentioned. In the case of aluminum powder, the experimental range was between 0.05 and 0.25 wt%; on the other hand, the percentages of hydrogen peroxide ranged from 0.5 to 4.0%. For characterization of the foamed geopolymer samples, quantitative X-ray diffraction (QXRD), thermogravimetric (TGA), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) techniques were used. The results of both foaming agents in zeolite-based geopolymers met Ecuadorian technical standards NTE INEN 643 and NTE INEN 638; nevertheless, the use of aluminum powder as foaming agent exhibited a better pore size distribution, suggesting a better performance. On the basis of these results, foamed geopolymers are suitable construction materials for the application of manufactured for masonry wall in houses.

1. Introduction

Nowadays, Portland cement constitutes one of the most in-demand construction materials, providing to our society the engineering material whereas our houses are built. However, this ever-increasing demand has brought some shortcomings that need

* Corresponding author at: Facultad de Ingeniería Mecánica y Ciencias de la Producción, Escuela Superior Politécnica de Litoral, ESPOL, Campus Gustavo Galindo km 30.5 Vía Perimetral, Guayaquil, Ecuador.
E-mail address: hbaykara@espol.edu.ec (H. Baykara).

https://doi.org/10.1016/j.conbuildmat.2018.03.253
0950-0618/

to be tackled. One of these problems is that its manufacturing process releases large amounts of CO_2 due to the decarbonation of limestone and the combustion of fossil fuels [1]. This environmental concern has driven the scientific community to evaluate possible alternative binders. One of the ecological-friendly alternatives is the use of geopolymers, increasingly developed since the early 1970s [2]. Geopolymers are environmentally friendly materials, and their manufacturing process may result in a meaningful reduction of CO_2 emissions compared to that of cement industry [3].

Among others, geopolymers as building materials possess excellent mechanical properties, fire and chemical resistance [4], conjointly with a rapid hardening, stability, and durability. Even geopolymers have been used in various technological applications such as aircraft interiors and automobiles, thermal shields for space shuttles, fire resistant compounds, or foam panels used as thermal insulation in the construction industry [2,5,6].

Foam type geopolymers have been developed to partially replace organic insulating materials like foamy polystyrene that causes serious health problems when it is burned. In order to succeed organic insulating materials, foamed geopolymers must exhibit high porosity and low density which makes these materials suitable for applications such as fire barriers and thermal insulation [4]. Porous foamed geopolymers are prepared by introducing a large volumetric fraction of air bubbles into the mixture when it is still in gel form. The formation of air bubbles is induced by adding a foaming agent like aluminum (Al) and zinc (Zn) powders [7] with alkali metal hydroxides, or hydrogen peroxide (H_2O_2) [4] during the mixing procedure.

Recently, novel foam type geopolymers, mainly using fly ash and slag as a solid precursor, have been reported as highly feasible alternatives to replace organic insulating materials [8–10]. During the synthesis of these foamed geopolymers, two types of foaming agents are used, i.e., hydrogen peroxide and aluminum powder. Comparing these two types of foaming agents within the same binder gel, Masi et at reported that aluminum powder provoked bigger pores than hydrogen peroxide in fly ash foamed geopolymer [1]. Regardless these effects in geopolymers, the authors also reported side effects during the foaming process that negatively affected the engineering performance of such a material. This suggests that an optimization of the amount of foaming agent is needed to be used in a scale-up process.

Natural aluminosilicates have also been used as the solid precursor in foam type geopolymer. One of the most used geological material is natural clay like kaolin and montmorillonite, which are usually thermally-treated to improve its reactivity [10,11]. Despite this widely and recently use of the natural aluminosilicate, the use of natural zeolite as the solid precursor in foam-type geopolymer synthesis is absent in the scientific literature. Therefore, this study can be considered as exploratory research, and as such, it explores the possibility of the use of natural zeolite, here in this study mostly mordenite, and shed light on foaming process in such geopolymers.

This study aims to prepare zeolite-based foamed geopolymers as replacement of prefabricated concrete slabs used for the external wall in buildings. As a solid precursor, these geopolymers used Ecuadorian natural zeolite-rich tuff containing quartz, mordenite, clinoptilolite, and calcite, located in the province of Guayas. Besides, this study explored the mix design of zeolite-based geopolymer and the effect of foaming agent on their compressive strength. the zeolite-based foam type geopolymers were characterized using quantitative X-ray diffraction (QXRD), scanning electron microscope/energy dispersive X-ray spectroscopy (SEM/EDS), thermal gravimetric analysis/differential scanning calorimetry (TGA/DSC), Fourier transform infrared spectroscopy (FTIR) techniques.

2. Materials and methods

2.1. Materials

Foam type geopolymers (FTG) were synthesized by using the alkaline solution of NaOH (Merck® 99% purity), sodium silicate (Merck®, d = 1.34 g/mL at 20 °C, 7.5–8.5% of Na_2O, 25.5–28.5% of SiO_2 and a SiO_2/Na_2O ratio of 3) and deionized water. The solid precursor, zeolitic-tuff was supplied by INDAMI S.A. which processes the tuffs from a Cayo formation (Coordinates: Latitude 2°09′06.2″S, Length 80°05′45.4″W). This zeolite-rich tuff exhibits a composition rich in mordenite, clinoptilolite, and quartz with the grain size of 28 µm (90% of the sample). The milled zeolitic tuff showed a median grain size (D_{50}) of 10.49 µm and (D_{90}) of 28.43 µm. Fig. 1. shows the cumulative and standard plot of de particle size distribution for zeolitic tuff after milling. The D_{50} and D_{90} values mean that 50% of the particles are smaller than the values corresponding to each point, respectively. The calcium hydroxide (Merck® 96% purity) is also used to be blended with zeolite-rich tuff (see Tables 1 and 2). As the foaming agents, aluminum powder (Lobachemie® 98% purity) and Fisher® hydrogen peroxide (30% dissolved in water) were used.

2.2. Methods

2.2.1. Specimen preparation

As previously reported, 10 M sodium hydroxide, sodium silicate and $Ca(OH)_2$ (3 wt% of the amount of zeolite) were used for the preparation of zeolite-based geopolymers [12]. Tables 1 and 2 depict the mix proportion of aluminum-foamed geopolymers, and hydrogen peroxide foamed geopolymer with activator/zeolite ratio of 0.6 and 0.4, respectively. The mixture of the reagents followed ASTM C305 [13], "Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency." After curing for 24 h at 60 °C, the demolded samples were aged for six days more at ambient condition. So, the samples were cured for seven days in total. The mechanical properties of the samples were tested through compressive strength. Some fractured pieces were collected to be used for the other characterization techniques.

The hydrogen and oxygen gases production via decomposition of hydrogen peroxide (H_2O_2) and aluminum powder reacting with the basic liquid matrix is presented in Fig. 2. [8,14,15]:

2.2.2. Particle size analysis

A Malvern® Mastersizer 2000 was used to analyze the particle size distribution of the zeolitic-tuff. In this study, Hydro 2000G wet cell of primary dispersion unit was used, using ethanol and taking values through a standard and cumulative distribution, where three measurements were done on average.

2.2.3. Compressive strength

A universal testing machine, Shimadzu® model UH-600KNI, was used to evaluate the mechanical performance of the foamed geopolymers after seven days of aging. The mechanical properties were tested basing on International Standard ASTM C 109, "Compressive Strength of Hydraulic Cement Mortars" [16], and then compared to NTE (Ecuadorian technical standard) INEN (Ecuadorian normalization institute) Standard 643 "Hollow Concrete Blocks, Requirements" standard [17] in order to evaluate if these geopolymers met the requirements.

2.2.4. Density

The density of the foamed geopolymers was analyzed using an OHAUS® EM-027 balance. The standard ($\rho = \frac{A}{A-B}(\rho_0 - \rho_L) + \rho_L$; the